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(19)

(54) COATING PROCESS

(71) We, LILLY INDUSTRIES LIMITED, a British Company, of Henrietta House, Henrietta Place, London, W.1., do hereby declare the invention for which we pray that a patent may be granted to us and the method by which it is to be performed, to be particularly described in and by the following statement:—

The invention relates to a method of preparing surface-coated particles.

In the past at least two methods have been used to coat particles, for example tablets for pharmaceutical use, with a suitable coating material. Evenly coated particles, however, have not always been obtained by these known coating processes. To obtain particles having an even surface coating it is desirable to change the orientation of the pellets so as to present each part of the surface of each particle to the coating material under similar conditions. The coating material must, of course, be applied in a manner complementary to the way in which the particles are presented to be coated.

In the pan coating method, which is one of the coating methods generally employed in the past, a rotating, elliptical, but nearly spherical, or cylindrical pan, is used. The particles move round with the circular motion of the pan until they tumble under gravity. The pan may, if desired, be provided with baffles. This method has a number of disadvantages. Firstly, "dead spots" may occur in the vessel where there is no movement of the particles. Additionally, there is poor overall mixing of the particles and the area exposed to the drying air or sprayed coating material is only slowly changing. In this method manual effort is necessary, for instance, to overcome "massing". The method is also of disadvantage in that the maximum rotation of the pan is determined by the centrifugal force which will overcome gravity and in that small particles are difficult to separate under gravity.

Fluidisation of particles in a stream of air within a vertical cylindrical or conical chamber, the particles being supplied with fluidising energy, usually by means of gas under a pressure which is less than that required for pneumatic transport and greater than that required for expansion of the bed, is the principle of another method already known. This method has the disadvantage that as the coating material is sprayed onto the particles, spray drying can occur before the coating material coats the particles. Additionally, careful control of fluidising parameters is required to prevent attrition between the particles. Furthermore, excessive air has to be used, resulting in inefficient use of heat. In this method, inefficient mixing of particles in the fluid bed also occurs. This results in an uneven application of coating.

According to the invention, there is provided a method for preparing surface-coated particles which method comprises rotating about a substantially vertical axis a frictional plate provided with a roughened, generally horizontal upper surface having a circular periphery closely adjacent an encircling, upwardly extending wall forming part of a vessel in which the plate rotates, particles to be coated being contacted with the upper surface of the plate and being propelled by centrifugal force across the upper surface of the plate and up the encircling wall of the vessel, and the particles being coated during their movement by a coating material supplied to the vessel.

The frictional plate may, for example, be rotated at a speed of from 40 r.p.m. to 1000 r.p.m. (a peripheral speed of about 50 m/min. to 1250 m./min. in the case of a circular having a diameter of 40 cm.) and, more commonly, at a speed of from 50 r.p.m. to 600 r.p.m.

As will be appreciated, the desired rate of rotation of the plate is dependent upon the size of the particles to be coated. It has been found, for instance, that a speed of rotation

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of about 400 r.p.m (peripheral speed of about 500 m./min. in the case of a 40 cm. diameter plate) is suitable for particles having a diameter of 1.5 mm. and that a rotational speed of about 200 r.p.m. (peripheral speed of about 250 m./min. for a 40 cm. diameter plate) is suitable for particles having a diameter of 6 mm.

The term "frictional plate" means a plate having a sufficiently frictional upper surface so as to cause particles placed thereon to move in a curved path towards the plate peripheral under the influence of centrifugal force generated by rotation.

The frictional upper surface may be achieved by perforation of the plate or by roughening of its upper surface. At least an annular portion extending to the periphery of the plate is perforated or roughened. Preferably, a solid frictional plate with a roughened upper surface is used. In a particular embodiment of the invention, the upper surface of the plate is formed with grooves, for instance 1 millimetre to 2 millimetres in depth and spaced apart at distances of from 2 millimetres to 4 millimetres, providing roughening of the surface. In one form of this embodiment, the member is formed with two sets of grooves, the grooves of the first set intersecting those of the second set preferably at right angles.

Alternatively, the roughening of the upper surface may be achieved by pitting the surface, for example by sand blasting.

The plate preferably is provided with an upwardly inclined or upwardly curved peripheral portion. In the former case the peripheral portion may be inclined to the general plane of the remainder of the upper surface of the plate at an angle of not more than 45°. In the case where the peripheral portion is upwardly curved, it is preferably in the form of a continuous curve, such as a circular arc, when the member is viewed in cross-section and as a whole. The curve blends smoothly with the remainder of the upper surface of the plate and preferably also with the upwardly extending wall of the vessel. Preferably, the peripheral portion, when upwardly curved, is curved to an extent such that a line extending from the inner edge to the outer edge of the portion is inclined at an angle of not more than 45° to the general plane of the remainder of the upper surface.

The method of the invention is applicable particularly to particles having a diameter of at least 0.15 mm. especially 0.25 mm. or more. The plate is therefore commonly sized so that in rotation the periphery of the upper surface of the plate clears the wall of the vessel by at least 0.15 mm. and, more commonly, by at least 0.25 mm.

In the most common construction of apparatus suitable for carrying out the method of the invention, the plate is mounted in the vessel at a position intermediate of the uppermost and lowermost extremities of the defining wall. Such a construction preferably includes means for providing, in the region of the vessel beneath the plate, a pressure higher than the pressure in the region above the plate in order to prevent slippage of the particles to be coated between the wall of the vessel and the periphery of the upper surface of the member. This particular feature of suitable apparatus is especially advantageous when the size of particles to be coated is less than the clearance between the periphery of the plate and the wall of the vessel. The means for providing the higher pressure may, for example, be in the form of a fan rotatably mounted in the region of the vessel beneath the plate. Alternatively, such means may be in the form of a duct connected between the lower region of the vessel and a source of pressure, such as a fan.

The coating material used to coat the particles may be supplied to the vessel, for example, in the form of an aqueous or evaporable non-aqueous solution of the material, in pulverulent solid form or in the form of a liquified, normally solid material. The coating material, when supplied in the form of an aqueous solution, is commonly a sugar or a synthetic polymer. When, as may be the case, the coating material is supplied in the form of its solution in a non-aqueous, evaporable solvent, the coating material is commonly a gum or a polymer. Specific examples of the coating material are given in the Table.

When the coating material is supplied in the form of a pulverulent solid, the particles to be coated are usually firstly wetted by means of a suitable liquid so that the pulverulent coating material adheres to the particles.

The coating material may be introduced into the vessel by means of simple pouring of the solid or liquid, or by means of a spray such as an airless spray. Often it is of advantage to introduce liquid coating materials in the form of a spray comprising finely atomized droplets.

TABLE

Aqueous sugar solution	Aqueous synthetic polymer solution	Non-aqueous gum or polymer solution	Dusted powdered solid	Liquefied solid
Sucrose, Maltose, or any solid-forming sugar.	Methylcellulose, Hydroxypropyl-methylcellulose, Polyvinyl alcohol.	Ethylcellulose, Shellac, Cellulose acetate phthalate.	Micro-crystalline cellulose, Starch, Talc, Magnesium stearate, calcium carbonate.	Beeswax, Polyethylene glycol.

A jet of air may be directed onto at least part of the upper surface of the plate whilst it is rotating. The jet of air may be at an elevated temperature which is preferably adjustable. This jet of air serves to expedite evaporation of the water or other evaporable solvent in cases where the coating material is supplied in the form of an aqueous or evaporable non-aqueous solution. When at lower temperatures the jet also serves to speed solidification of coatings on particles when the coating material is supplied as a liquefied solid. Conveniently, the jet is directed onto only a quadrant portion of the upper surface of the rotating plate.

In some cases it is preferable to heat the particles prior to introduction to the vessel. For example, particles to be coated with a material which is supplied to the vessel in the form of an aqueous solution are preferably raised to a temperature of from 90°F to 100°F. The preheating of the particles may be carried out by means of hot air treatment in a suitable vessel or by thermal preconditioning in a hot room.

The construction and operation of two examples of apparatus suitable for carrying out the method of the invention will now be described in more detail with reference to the accompanying drawings in which:

Figure 1 is a cross-sectional view of one embodiment of an apparatus suitable for carrying out the method of the invention; and

Figure 2 is a cross-sectional view of part of an apparatus similar to that of Figure 1 but having a frictional plate with an upwardly curved upper surface.

Referring to Figure 1 of the drawings, the apparatus shown comprises a cylindrical vessel 2 having smooth internal walls 3, and an open top 4. The vessel has an internal diameter of from 30 cm. to 100 cm., commonly 40 cm. Rotatably mounted at or near the base is a circular plate 5, having a generally flat, rough surface milled with grooves. The grooves, which are in the form of two sets those of one set intersecting at right angles with those of the other, are 1 to 2 mm. deep and spaced apart at distances of from 2 to 4 mm. At least an annular portion extending to the periphery of the plate is roughened. The plate is driven by a motor (not shown) connected by means of a pulley to a pulley wheel 6 rigidly connected to the shaft 7 which is rigidly secured to the plate 5 by means of a screw 8. The shaft 7 is located in a housing 9 and is supported by ball bearings 10*a* and 10*b*, 11*a* and 11*b*, and 12*a* and 12*b*, (not all bearings shown). The bearings are all lubricated for example by oil or grease. Seals 13*a* and 13*b*, 14*a* and 14*b*, and 15*a* and 15*b*, are provided to retain the lubricant in the region of the bearing surfaces. The seals 13*a* and 13*b* are located in the structure of a scraper 16 which scrapes the floor of the vessel 2, while the seals 14*a* and 14*b* are located in a top bearing cover 17. The oil seals 15*a* and 15*b* are located between the surface of the shaft 3 and a lower bearing cover 18. Grease nipples 19 and 20 serve for supplying lubricant to the bearings.

An angled pipe 21 for supplying a jet of air onto the upper surface of the plate extends horizontally towards the centre of the vessel 2 and then deviates to an angle of 90° so that its outlet end 21*a* is directed towards a quadrant surface of the plate 5. A volume controlling means (not shown) is associated with the pipe to adjust the volume of air supplied. A means (not shown) of controlling the temperature of the air, such as a form of heating located at the source, is also provided.

A coating material inlet assembly 22 is located diametrically opposite the point at which the pipe 21 penetrates the walls 3 of the vessel. As shown, the inlet 22 comprises two separate components 22*a* and 22*b* for independently supplying solid and liquid material to the vessel.

An air pressure supply 23 is provided for introducing a stream of air to the region beneath the plate 5. The supply 23 comprises a duct having a portion 24 provided with a group 25 of perforated plates located across its diameter, a portion 26 terminating at an aperture 27, and a source (not shown)

of air pressure, such as a fan.

A removal aperture 28, closable by a plate-like lid 29 operable by a handle 30, is provided through which the coated particles may be removed.

The apparatus, part of which is shown in Figure 2, is similar to that shown in Figure 1 and like parts are designated by like reference numerals. The plate 5 of the apparatus of Figure 2, however, is provided with an upwardly curving peripheral portion 5*a*. Such an apparatus is described and claimed in the Complete Specification of our copending cognate Patent Applications Nos. 29752/70 and 11522/72 (Serial No. 1346416).

The particulate material, the particles of which will form the cores of the resulting surface-coated particles, are suitably introduced into the vessel 2 by means of a hopper or bowl, for instance, one 35 mm. in diameter.

In operation of the apparatus just described the particles in contact with the surface of the plate are urged outwardly across the roughened surface of the plate by centrifugal force and are deflected upwardly before falling back onto the upper surface of the plate to be thrown outwardly again.

The following is an illustrative example of a method according to the invention:

2.5 kgm. of particulate material, the particles of which were generally spherical having a diameter of about 1.5mm., were preheated in a vessel by means of hot air to a temperature of 90°F to 100°F. The so treated particles were then quickly placed on the upper surface of a frictional plate in an apparatus of the kind comprising a cylindrical vessel having an internal diameter of about 40 cm. The plate was then rotated at a generally constant rate of approximately 400 r.p.m. and finely atomised droplets of an aqueous sugar solution were introduced into the vessel. A jet of air was blown down onto a quadrant surface of the plate and the process was continued until such time as a desired coating had been formed on the particles. The particles were then removed.

It will be appreciated from the foregoing description that the invention provides a method in which the particles are rapidly coated, due to high speed of motion. The particles may be relatively small due to the stronger centrifugal separative forces. It will also be appreciated that the particles move in a regular pattern and, since the particles are always in motion relative to each other, massing does not tend to occur. More rapid drying may also be achieved.

WHAT WE CLAIM IS:—

1. A method for preparing surface-coated particles which method comprises rotating about a substantially vertical axis a frictional plate provided with a roughened, generally horizontal upper surface having a circular periphery closely adjacent an encircling, upwardly extending wall forming part of a vessel in which the plate rotates, particles to be coated being contacted with the upper surface of the plate and being propelled by centrifugal force across the upper surface of the plate and up the encircling wall of the vessel and the particles being coated during their movement by a coating material supplied to the vessel.

2. A method as claimed in claim 1 wherein the plate is rotated at a speed of from 40 r.p.m. to 1000 r.p.m.

3. A method as claimed in claim 1 or claim 2 wherein the plate is rotated at a speed of from 50 r.p.m. to 600 r.p.m.

4. A method as claimed in any one of claims 1 to 3 wherein the encircling wall of the vessel clears the periphery of the upper surface of the plate by at least 0.15 mm.

5. A method as claimed in any preceding claim wherein the encircling wall of the vessel clears the periphery of the upper surface of the plate by at least 0.25 mm.

6. A method as claimed in any preceding claim wherein the upper surface of the plate is formed with grooves.

7. A method as claimed in any one of claims 1 to 6, wherein the upper surface of the plate is formed with first and second sets of grooves, the grooves of the first set intersecting with those of the second set.

8. A method as claimed in claim 6 or claim 7 wherein the grooves are spaced apart by distances of from 2 to 4 mm.

9. A method as claimed in any one of claims 6 to 8 wherein the grooves are from 1 to 2 mm. in depth.

10. A method as claimed in any preceding claim wherein the plate is mounted in the vessel intermediate the uppermost and lowermost extremities of its defining wall.

11. A method as claimed in claim 10 wherein the air pressure in the region of the vessel below the plate is maintained at a higher value than in the region of the vessel above the plate in order to prevent passage beneath the plate of particles having a diameter of less than the clearance between the adjacent wall of the vessel and the part of the plate nearest thereto.

12. A method as claimed in any preceding claim wherein the vessel is of cylindrical form having a uniform circular cross-section having a diameter of from 30 cm. to 100 cm.

13. A method as claimed in any preceding claim wherein the coating material is supplied to the vessel in the form of a solution of the coating material in an evaporable solvent.

14. A method as claimed in claim 13 wherein the said solution is an aqueous solution of a sugar, methylcellulose,

hydroxypropylmethylcellulose or polyvinyl alcohol.

15. A method as claimed in claim 13 wherein the said solution is a solution of ethylcellulose, shellac or cellulose acetate phthalate in a non-aqueous solvent.

16. A method as claimed in any one of claims 1 to 12 wherein the coating material is supplied to the vessel in pulverulent solid form and the particles to be coated are wetted prior to supply of the coating material.

17. A method as claimed in claim 16 wherein the coating material is microcrystalline cellulose, starch, talc, magnesium stearate or calcium carbonate.

18. A method as claimed in any one of claims 1 to 12 wherein the coating material is supplied in the form of a liquefied, normally solid material.

19. A method as claimed in claim 18 wherein the coating material is beeswax or polyethyleneglycol.

20. A method as claimed in any preceding claim wherein the coating material is supplied in the form of an airless spray.

21. A method as claimed in any preceding claim wherein the coating material is supplied in the form of finely atomised droplets.

22. A method as claimed in any preceding claim wherein a jet of air is directed onto at least part of the upper surface of the rotating plate.

23. A method as claimed in claim 22 wherein the jet of air is at an elevated temperature.

24. A method as claimed in claim 22 or claim 23 wherein the jet of air is directed onto only a quadrant portion of the upper surface of the plate.

25. A method as claimed in any preceding claim wherein the plate is provided with an upwardly curved or inclined peripheral portion.

26. A method as claimed in claim 1 and substantially as hereinbefore specifically described with reference to either of the drawings.

27. A method as claimed in claim 1 and substantially as hereinbefore specifically described in the Example.

28. Surface-coated particles, whenever prepared by the method claimed in any one of the preceding claims.

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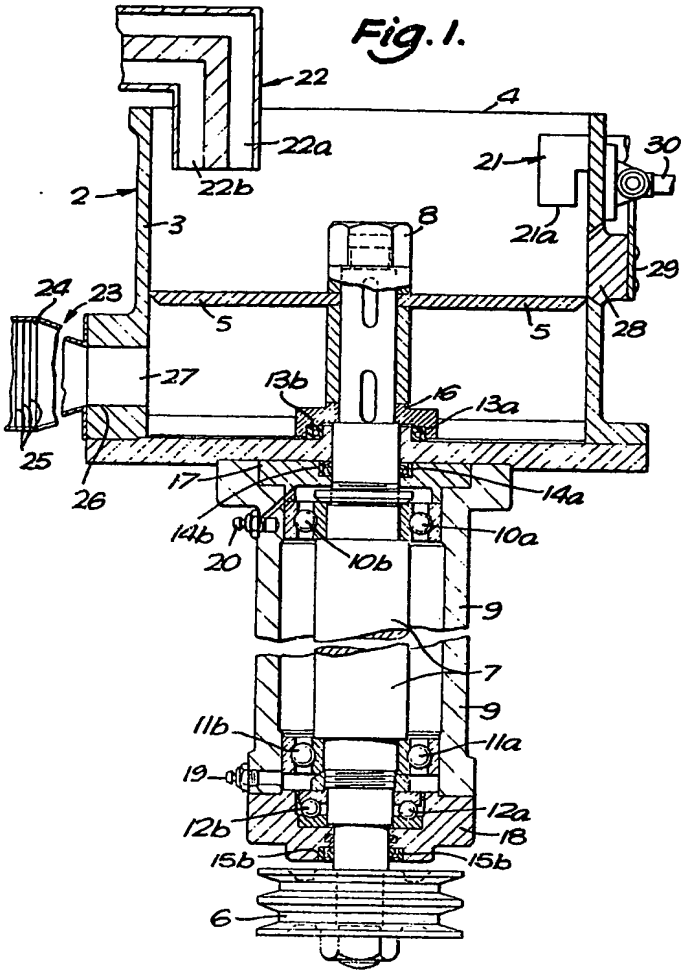
Fig. 1.



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Sheet 2

Fig. 2.